

# PATENT SPECIFICATION

NO DRAWINGS

1,121,563



1,121,563

Inventor: MONROE LANZET.

Date of Application and filing Complete  
Specification: 24 May, 1966.

No. 22999/66

Complete Specification Published: 31 July, 1968.

© Crown Copyright, 1968.

Index at Acceptance:—C4 X11; A5 B31.

Int. Cl.:—B 05 b 17/00.

## COMPLETE SPECIFICATION.

### Self-propelled Homogeneous Liquid Compositions

We, YARDLEY AND COMPANY LIMITED, a British Company of 33 Old Bond Street, London, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates to the production of self-propelled homogeneous liquid compositions which when dispensed from suitable aerosol containers produce foams of transient stability.

There have been proposed and used, aerosol foam compositions for cosmetic purposes. While such compositions have been stated to be suitable for use in transparent containers or dispensers, they have had the disadvantage in that at low ambient temperatures some of the constituents separated out. They formed at the bottom of the aerosol an unsightly heterogeneous mass or precipitate. Such temperatures below 60°-65° F., and often as low as 45° F. are often encountered in the bathroom in mornings of low outside temperatures. These precipitates, usually of the surfactants present, were frequently so serious that foaming has even been prevented at such low temperatures.

The present invention is intended and adapted to overcome the defects in the prior art, it being among the objects of the invention to provide an aerosol foam composition which remains clear at said low temperatures and prevents any noticeable amount of precipitation in the container.

Compositions according to the invention can be made up which will not cause stinging of the skin, avoid producing red blotches on the skin and which will not cause irritation thereof.

The constituents and the proportions thereof can be so chosen that the foam pro-

duced therefrom will be self-collapsing in a very short time.

The invention is directed to the production of such fluid compositions at low pressures and with clarity at extremely low ambient temperatures so that the product will remain clear and fluid over a wide temperature range and thus be esthetically suitable for packaging in clear glass, plastic coated glass and clear plastic aerosol containers. Body fresheners, after shave lotions, hair grooms, and other similar quick breaking foams are made considerably more elegant and desirable when dispensed from a clear single phase aerosol package.

Part of the problem has been restricting surfactant selection primarily to certain commercially available blended surface active agents. In order to achieve low temperature clarity, applicants have had to develop their own special blends of suitable surfactants in addition to making other modifications in composition which will be more fully described later.

#### SKIN STINGING AND IRRITATIONAL POTENTIAL

Applicants' work in this area has shown that high alcohol levels (i.e., 55-65%) are quite irritating to the intact skin often causing red blotches upon second application. Since many cosmetic applications of these foams are on the skin, this is quite undesirable. This effect is even more pronounced in after-shave foams which are applied to abraded skin.

For these reasons Applicants have developed foams with ethanol contents as low as 32% of the 95% alcohol, or even lower in some cases. Even non-irritating alcohol levels may sting the face and therefore cosmetic elegance demands foams of negligible stinging potential. Collapsible foam products of the present invention formulated with

alcohol levels in the range of 30-45% alcohol possess no or minimal stinging attributes.

#### FOAM DURATION AND COLLAPSIBILITY

- 5 Another aspect of the present systems is the stability of the foam puff. Prior art compositions were often times too stable. As distinctly different from the prior art, Applicants have found that suitable foams should  
10 be of the self-collapsing type with durations of from 10 to 60 seconds. Consumers have actually shown a preference for this type since more stable foams resist rubbing and are often-times extruded between the fingers  
15 when handled. The foam puff characteristics are controlled by the concentration and composition of the surfactant blend, the concentration of the alcohol, and the type and concentration of propellants selected.

#### 20 INGREDIENTS

The essential basic ingredients of a quick-breaking foam of transient stability are as follows:

- 25 A—A blend of alcohol (30 to 55%) and water to act as the vehicle.

B—A blend of surfactants at concentrations from 0.4 to 5.0% by weight.

- 30 C—Either one or a blend of compatible hydrocarbon or halocarbon gases at concentrations from 2 to 18% by weight.

Additionally, any useful auxiliary diluent might be employed which would not interfere with the clarity or the foam of the finished aerosol composition.

#### 35 A—The Alcohol/Water Ratio

- The surfactants normally used in these aerosols are chosen to be soluble in the alcohol and insoluble in water. The water/alcohol concentration ratio is then adjusted  
40 at ambient temperature to the point at which the surfactant blend is just barely insoluble in the mixture. The temperature at which the concentrate will clear and become homogeneous usually coincides with the upper  
45 temperature at which a usable foam may be generated when the concentrate is dispensed as an aerosol.

The expression "alcohol" refers to ethanol, methanol, isopropanol or n-propanol.

- 50 When lower alcohol levels are used, stinging of the skin is reduced, therefore Applicants have endeavored to employ as little alcohol as possible in the composition concomitant with clarity of the finished  
55 aerosol. This is accomplished by:

1. Selection of surfactants with good hydro-alcoholic solubility.
2. Use of glycols and ethers as couplers.
3. Increased propellant concentration.

#### 60 B—Surfactant Selection

- The character of the foam and its stability are most directly affected by the concentration and choice of surfactant. In no case however, should it be necessary to use more  
65 than 5% by weight of surfactant in the total

aerosol because additions in excess of this quantity produce tackiness, and insulated feeling on the skin and are only really acting as solubilizing replacements for alcohol. This replacement for alcohol may be more  
70 elegantly achieved by using more propellant, glycol, or water, or a combination of these, thus avoiding deposition of excessive solids on the skin. Suitable surfactant blends are somewhat limited because they must be  
75 solid substances, clearly soluble in the finished aerosol composition including propellants down to about 6° C., but insoluble in the concentrate (all the remaining ingredients except propellants) at temperatures  
80 as high as 30° C., or even higher, if foam is to be generated at temperatures higher than 30° C.

Suitable surfactant blends for best clarity combine C<sub>12</sub>-C<sub>22</sub> saturated fatty alcohols with 85 nonionic surfactants chosen from the class consisting of polyoxyethylene derivatives of said fatty alcohols, fatty acids and lanolin acids and alcohols, prepared by reacting a  
90 suitable alcohol or acid with ethylene oxide or polyethylene oxide. Some typical examples of these blends are as follows:

	% by Wt.	
a) Cetyl Alcohol	25-60	
pentaethoxy (5 moles ethylene oxide) C <sub>16-20</sub> Fatty Alcohol ether	75-40	95
b) Cetyl Alcohol	33-66.0	
diethoxy Stearyl ether	53-20	
deca ethoxy Stearyl ether	20-14	
c) Cetyl Alcohol	50.0	100
polyethylene glycol (40 moles ethylene oxide) lanolin alcohol ether	50.0	
d) diethoxy Stearyl ether	28-35	
Cetyl Alcohol	3- 6	105
Polyethylene Glycol (25 moles ethylene oxide) lanolin alcohol ether	58-66	
e) Arachidyl Alcohol	6.0	
Polyoxyethylene (40 moles ethylene oxide) lanolin alcohol ether	71.0	110
Diethoxy Stearyl ether	23.0	
f) diethoxy Stearyl ether	54-64	
Cetyl Alcohol	46-36	115

Blends of nonionic surfactants based upon ethers of sorbitan fatty acid esters, ethoxylated, saturated C<sub>16-20</sub> fatty alcohols and ethoxylated lanolin alcohols have also been found to be extremely effective surfactants  
120 when good foam and low temperature clarity is desired in systems containing lower alcohol levels. Examples of such systems are as follows:

	% by Wt.	
g) Any or a blend of Pentaethoxy-decaethoxy Cetyl/Stearyl/Arachidyl ethers	100.0	
h) Diethoxy Stearyl Ether	25-48	
Polyoxyethylene (40 moles		130

- ethylene oxide) Lanolin  
alcohol ether 75-52  
i) Tetra ethoxy Stearyl ether 25  
Polyoxyethylene (20 moles  
ethylene oxide) Sorbitan  
5 monolaurate 75  
j) tetraethoxy Cetyl/Stearyl ether 35-40  
decaethoxy Cetyl/Stearyl ether 65-60

#### C—The Propellants

- 10 The solubility of propellants in high water systems is a limitation on the propellant concentration. To overcome this limitation we select propellants which have the highest relative water solubility. Ex-  
15 amples of some of these propellants are butane, monochloro-difluoro-ethane, difluoro-ethane, trichloro-monofluoro-methane and dichloro-monofluoro-methane which help to increase propellant compatibility  
20 with the concentrate.

These more soluble propellants may often-times be used with other less soluble gases such as dichloro-difluoro-methane and tetrafluoro-dichloro-ethane to derive the best  
25 characteristics of both types.

- All other things being equal, the maximum concentration of propellant which the system can maintain in a single liquid phase is dependent upon the level of water in the  
30 aerosol. Less water in the concentrate allows more propellant to be dissolved but the foam characteristics are also dependent upon the water, and poor foam generation results if the water concentration is reduced  
35 below 20% unless high glycol levels (above 10%) are utilized to make up for the lower water percentage.

The lowest pressures are obtained in low water systems containing monochloro-difluoro-ethane or a combination of trichloro-monofluoro-methane or tetrafluoro-dichloro-ethane in about equal proportions with said monochloro-difluoro-ethane. Low pressures may also be achieved with blends of  
45 chloro-difluoro-ethane with trichloro-monofluoro-methane or tetrafluoro-dichloro-ethane containing 30% or less by weight of dichloro-difluoro-methane. The pressures which are preferred range from about 15 to  
50 25 psig, but under some conditions pressures may range from 10 to 40 p.s.i.g.

Propellant solubility is also critical because foam stability is a function of the solubility of the propellant in the aqueous  
55 alcohol solution.

#### D—Auxiliary Ingredients (Couplers, diluents, germicides, etc.)

While the basic essential ingredients of these improved foams have been outlined as  
60 consisting of alcohol, water, propellants and a nonionic surfactant blend, other useful ingredients such as glycols, fatty ethers, and fatty esters may be included as additional couplers or emollients. Certain liquid surf-  
65 actants such as di- and tri-ethoxy oleyl ether

and tetra-ethoxy lauryl ether, though not structural foaming agents, assist in produc-  
1,121,563

ing clarity down to lower temperatures or permit use of higher propellant levels than 70 would otherwise be attainable.

Healing agents, bacteriostatic agents and other medicaments and emollients may also be included for their utility but do not constitute a critical part of the present inven-  
75 tion. In any case low stinging auxiliary agents such as these may also be selected and anti-irritants and anti-inflammatories such as polyvinyl pyrrolidone or guaiazulene might also be included. 80

The following specific examples are illustrative of the invention although not limiting the scope thereof:

#### Example 1

	% by Wt.	
95% Ethanol	32.60	
Surfactant blend "g"	0.40	
Glycerine	10.00	
Water	56.81	
Cetyl trimethyl ammonium bromide	0.05	90
Perfume	0.14	

When two grams of difluoro-ethane was pressure filled into the bottle containing this concentrate it cleared and became homo-  
95 geneous. The finished aerosol, fitted with a 95 foam actuator, produced a good tight bubbled quick-breaking after-shave foam which caused no stinging even when repeatedly applied to the face. The finished aerosol was clear at temperatures of as low  
100 as 6° C.

#### Example 2

	% by Wt.	
(95% Alcohol	51.0	
(		105
(Surfactant Blend "b"	2.7	
(		
C (Menthol	0.1	
(		
(Perfume	0.8	110
(		
(Hexachlorophene	0.1	
(		
(Distilled Water	34.9	
(Trichloro-monofluoro-methane	4.4	115
P (		
(Monochloro-difluoro-ethane	6.1	

The above after-shave foam concentrate ingredients (C) were blended together to form a uniform gelled slurry and filled into clear  
120 aerosol bottles. After purging to remove air the bottles were sealed with a regular glass bottle aerosol valve. When the propellant mixture (P) was added through the valve, the finished aerosol became a homogeneous  
125 single liquid phase clear down to about 6° C. The pressure of the finished aerosol was 20 psig  $\pm$  2 psig at 70° F. When applied to the face after shaving, the foam produced no irritation and only the desired  
130

minimal momentary stinging sensation.

#### Example 3

A medicated body freshener foam was prepared according to the formula shown below, introduced into a clear aerosol bottle and sealed with a standard glass bottle valve after first purging to remove air. The heterogeneous concentrate was clarified by the addition of the propellant phase (P). The finished aerosol had a pressure of 25 psig  $\pm$  2 psig at 70° F. and was clear down to about 6° C.

When fitted with a standard foam actuator, a foam puff dispensed from the aerosol had desirable foam body and texture and a stability of from 30 to 60 seconds at ambient temperature.

When applied to the skin, no irritation or stinging was produced even after repeated re-application.

	% by Wt.
(95% Ethanol	38.70
(Surfactant Blend "a"	1.90
(Hexachlorophene	0.30
(Polyethylene Glycol 200	9.70
(Perfume	0.2
(Distilled Water	45.9
P Monochloro-difluoro-ethane	3.3

#### Example 4

The following medicated skin product concentrate (C) was prepared as in previous Examples and was a semi-solid opaque mass at 70° F. Addition of the propellant blend as before resulted in a clear homogenous aerosol with a pressure of only 18 psig  $\pm$  2 psig at 70° F. The entire aerosol packed in a clear uncoated bottle was clear at temperatures down to 6° C.  $\pm$  1° C. The foam puff was particularly fine bubbled and emollient due to the presence of a somewhat higher concentration of surfactant blend.

	% by Wt.
(95% Ethanol	47.8
(Surfactant Blend "d"	2.9
(Water	38.6
(N-lauryl-colamino formyl methyl pyridinium chloride	0.1
(Perfume	0.9
(Allantoin	0.1
(Trichloro-monofluoro-methane	4.4
P (Monochloro-difluoro-ethane	6.1

Among the advantages inherent in the present invention are the absence of stinging

of the skin upon repeated application of the foam aerosol, the absence of irritation of even freshly shaved skin, and the avoidance of formation of red blotches on the skin. An important advantage lies in the clarity of the aerosol at temperatures as low as 6° C. This is obtained by specially selected blends of surfactants, lowering of the alcohol content, increase of the propellant content, and selection of propellant blend.

#### WHAT WE CLAIM IS:—

1. Low pressure aerosol foam compositions consisting of a vehicle, a surfactant blend and a propellant; said vehicle being a monoalcohol having 1 to 3 carbon atoms in the amount of 30 to 55% and water in sufficient amount in the concentrate to make 100%; said surfactant blend being present in amount of 0.4 to 5.0%, soluble at temperatures of about 6° C., in said composition, and being comprised by at least one of the fatty alcohols having 12 to 22 carbon atoms combined with a nonionic surfactant which is a polyethylene oxide derivative of said fatty alcohols, of a fatty acid, of a lanolin acid or of a lanolin alcohol; said propellant being present in amounts of 2 to 18%, and being butane, monochloro-difluoro-ethane, difluoro-ethane, trichloro-monofluoro-methane, dichloro-monofluoro-methane, dichloro-difluoro-methane or tetrafluoro-dichloro-ethane, the pressure thereof being 10 to 40 p.s.i.g. at ambient temperatures.

2. Aerosol foam compositions according to claim 1 in which said surfactant includes ethers of sorbitan fatty acid esters, and ethoxylated, saturated C<sub>16-20</sub> fatty alcohols and ethoxylated lanolin alcohols.

3. Aerosol foam compositions according to claim 1 in which the water-alcohol ratio is such that at ambient temperatures said surfactant blend is barely soluble therein.

4. Aerosol foam compositions according to claim 1 in which the amount of water is at least 20%.

5. Aerosol foam compositions according to claim 1 in which there is present also as coupler a substance selected from the class consisting of glycols, fatty ethers and fatty esters.

6. Aerosol foam compositions according to claim 1 in which said aerosol foam breaks in about one minute.

7. Aerosol foam compositions according to claim 1 in which said aerosol foam puffs over a range of about 6°-30° C.

8. Aerosol foam compositions according to claim 1 in which the propellant is a mixture of monochloro-difluoro-ethane with trichloro-monofluoro-methane in about equal proportions.

9. Aerosol foam compositions according to claim 1 in which the ratios of water/alcohol/propellant/surfactant are such that

said foam collapses on the skin without application of pressure by the temperature of the skin.

10. Aerosol foam compositions according to claim 1 in which the following approximate composition is used:

	% by Wt.
Alcohol	32.60
Penta-ethoxy to decaethoxy-	
10 Cetyl/Stearyl/Arachidyl ethers	0.40
Water	57.00
Glycerine	10.00 , and
difluoro-ethane.	

11. Aerosol foam compositions according to claim 1 in which the following approximate composition is used:

	% by Wt.
Alcohol	51.0
Cetyl/alcohol/diethoxy stearyl	
20 ether/decaethoxy stearyl ether	2.7
Water	34.9 , and
trichloro-monofluoro-methane with mono-	
chloro-difluoro-ethane.	

12. Aerosol foam compositions according to claim 1 in which the following approxi-

mate composition is used:

	% by Wt.
Alcohol	38.70
Cetyl alcohol/pentethoxy fatty	
alcohol ether	1.90 30
Water	45.90
Polyethylene glycol 200	9.70 , and
monochloro-difluoro-ethane.	

13. Aerosol foam compositions according to claim 1 in which the following approximate composition is used:

	% by Wt.
Alcohol	47.8
Diethoxy stearyl ether/cetyl	
alcohol/polyethylene (25) glycol	
lanolin alcohol ether	2.9 40
Water	38.6 , and
trichloro-monofluoro-methane with mono-	
chloro-difluoro-ethane.	

For the Applicants:—  
WILSON GUNN & ELLIS,  
Chartered Patent Agents,  
57 Market Street,  
Manchester, 1.

Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd., Berwick-upon-Tweed, 1968.  
Published at the Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.

